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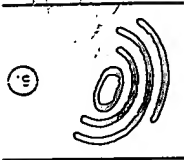
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(54) Preparation of polymer foam and product.

(57) A method for producing a thermoplastic polymer extruded foam body having an average cell size of from 0.05 mm to 3.5 mm, a density of from 1.0 lbs./ft³ (16 kg/m³) to 5.0 lbs./ft³ (80 kg/cm³), a minimal cross-sectional thickness of 0.5 in (1.3 cm) and a minimal cross-sectional area of 8 in² (52 cm²) comprises the steps of: heat plastifying the resin; introducing the plastified resin into a mixing device; introducing a blowing agent into the mixing device; maintaining a pressure in the mixing device at or above a pressure greater than an equilibrium vapor pressure of the blowing agent in the resin and blowing agent mixture; passing the mixture through a cooling device; passing the mixture through a die having a given die pressure greater than atmospheric pressure; maintaining a specific defined minimum critical pressure drop between the pressure at the inlet of the mixing device and the inlet of the die. Blowing agents useful in such process are disclosed as well polymer foam bodies made by the process and consistently having improved uniformity of surface quality.



European
Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 10 6475

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	US-A-4 222 729 (RAGAZZINI ET AL) - - -	1-4	B 29 C 67:22 C 08 J 9:12 C 08 J 9:14
A	US-A-4 222 729 () * column 1, line 12 - line 48; figure 1 *** column 4, line 45 - column 5, line 49 *** column 6, line 61 - line 68 *** column 9, line 33 - line 37; example 1 ** - - -	7-11	
X	DD-A-114 926 (LAUTERBERG) - - -	1-4,10	
Y	DD-A-114 926 () * page 3, column 1, line 22 - line 38 ** - - -	5-9,11-12	
Y	US-A-4 387 169 (ZABROCKI) * abstract; claims; examples ** - - -	5-9,11	
Y	EP-A-0 079 012 (MARYLAND CUP CORP) - - -	11-12	
A	EP-A-0 079 012 () * page 3, line 11 - line 30; claims 1-2.28-32; examples ** - - -	1-4,10	
A	US-A-3 300 554 (BACHUS) * column 3, line 14 - line 16; claims; figures 3B,7 *** column 7, line 21 - line 32 ** - - -	1-3,11	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	US-A-4 071 591 (KOBAYASHI ET AL) * column 4, line 14 - line 61 ** - - -	1-4,11	B 29 C
A	US-A-4 613 471 (HARRIS) * column 7, line 63 - column 8, line 39; claims 1-17; figure 1 * - - -	1-12	
A	US-A-3 972 970 (TAYLOR) * column 6, line 31 - column 7, line 68 *** abstract ** - - - -/-	1-2,11-12	
The present search report has been drawn up for all claims			
Place of search		Date of completion of search	Examiner
The Hague		15 November 91	PIPPING L.E.L.
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O: non-written disclosure		&: member of the same patent family, corresponding document	
P: intermediate document			



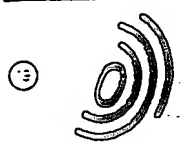
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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	US-A-3 817 669 (BUCKNER) * claims; figure 2: examples 1-2 ** -----	1-12	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
The present search report has been drawn up for all claims			
Place of search		Date of completion of search	Examiner
The Hague		15 November 91	PIPPING L.E.L.
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X: particularly relevant if taken alone</p> <p>Y: particularly relevant if combined with another document of the same category</p> <p>A: technological background</p> <p>E: earlier patent document, but published on, or after the filing date</p> <p>D: document cited in the application</p> <p>L: document cited for other reasons</p> <p>** member of the same patent family corresponding</p>			



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Preparation of polymer foam and product.

A method for producing a thermoplastic polymer extruded foam body having an average cell size of from 0.05 mm to 3.5 mm, a density of from 1.0 lbs/ft³ (16 kg/m³) to 5.0 lbs/ft³ (80 kg/cm³), a minimal cross-sectional thickness of 0.5 in (1.3 cm) and a minimal cross-sectional area of 8 in² (52 cm²) comprises the steps of: heat plastifying the resin; introducing the plastified resin into a mixing device; introducing a blowing agent into the mixing device; maintaining a pressure in the mixing device at or above a pressure greater than an equilibrium vapor pressure of the blowing agent in the resin and blowing agent mixture; passing the mixture through a cooling device; passing the mixture through a die having a given die pressure greater than atmospheric pressure; maintaining a specific defined minimum critical pressure drop between the pressure at the inlet of the mixing device and the inlet of the die. Blowing agents useful in such process are disclosed as well polymer foam bodies made by the process and consistently having improved uniformity of surface quality.

EP 0 445 847 A2

Such foam preparation is set forth in U.S. Patent Nos. 4,393,016 and 4,451,417, respectively. An alternative blowing agent system utilizing carbon dioxide and an alkane is set forth in U.S. Patent Nos. 4,344,710 and 4,424,287.

Alkenyl aromatic polymer foam has also been prepared as set forth in U.S. Patent No. 4,636,527, using as a blowing agent a mixture of carbon dioxide, ethyl chloride and optionally a fluorocarbon member selected from dichlorodifluoromethane, 1-chloro-1,1-difluoroethane and mixtures of these fluorocarbons.

It would be desirable if there were available a process for the preparation of alkenyl aromatic polymer foam which did not cause blow holes, poor skin quality and gassing at the die.

It would also be desirable if in the process of the present invention there was produced a novel alkenyl aromatic polymer foam prepared from more environmentally acceptable blowing agents.

These benefits and other advantages in accordance with the present invention are readily achieved in a process for producing an alkenyl aromatic synthetic resin extruded foam body having closed cells with an average cell size of from about 0.05 millimeter (mm) to about 3.5 mm, a density of from about 1.0 pound per cubic foot (pcf) (16kg/m^3) to about 0.5 pcf (80kg/m^3), a minimal cross-sectional thickness of one half (1/2) inch (1.3cm) and a minimal cross-sectional area of eight (8) square inches (52cm^2) including the steps of: (a) heat plastifying the alkenyl aromatic synthetic resin; (b) introducing the plastified resin and a blowing agent into a mixing device having an inlet maintained at a pressure, P_M , which is greater than an equilibrium vapor pressure of the blowing agent in the alkenyl aromatic synthetic resin and blowing agent mixture; (c) passing the mixture through a cooling device; (d) passing the cooled mixture through a die having a die inlet pressure, P_D , which is greater than atmospheric pressure; all wherein the quality of the foam's surface is controlled by deliberately maintaining the pressure drop from the mixer's inlet to the die's inlet, ΔP , at a pressure drop greater than or equal to an empirically predetermined minimum and critical pressure drop, ΔP_C , for the given mixture of resin and blowing agent.

Also contemplated within the scope of the present invention is an alkenyl aromatic synthetic resin extruded foam prepared from known blowing agents in accordance with the method of the present invention.

Further contemplated as within the scope of the present invention is an alkenyl aromatic synthetic resin extruded foam prepared from more environmentally acceptable blowing agents in accordance with the method of the present invention, which foams have good to excellent surface quality as measured by a test given hereinafter.

Still further contemplated within the scope of the present invention are polystyrene extruded foams prepared in accordance with the present invention.

Yet further contemplated within the scope of the present invention are styrene/acrylic acid copolymer extruded foams prepared in accordance with the present invention.

Still yet further contemplated within the scope of the present invention are ionomeric styrene/acrylic acid copolymer extruded foams prepared in accordance with the present invention.

For decades prior to this invention was made, it had been believed that as long as a die pressure is maintained above a vapor pressure of blowing agent systems at a given foaming temperature, it is possible to produce good quality closed cell low density foams with a good skin quality. Commercial foams prepared from current methods sometimes contain different amounts of blow holes and skin cracks or textures. Furthermore, it is difficult to produce a low density extruded foam with blowing agent systems containing environmentally acceptable blowing agents such as methane, ethane, carbon dioxide, nitrogen, water, and certain fluorocarbons and chlorofluorocarbons containing hydrogen.

In marked contrast to the prior art, foams prepared in accordance with the present invention can "consistently" have a low density extruded foam with improved skin quality and physical properties. The present invention also reduces the scrap rate resulting in a better utilization of raw materials, cost savings, and less emission of volatile organic compounds to the atmosphere.

Figures 1-19 are self-explanatory schematic drawings of various processes according to the invention involving measurement of the pressure drop from the mixer's inlet to the die's inlet.

Also, in contrast to the prior art, the present invention provides an "early warning" signal of deterioration in extrusion conditions before the deterioration is so great that it actually causes blow holes in the surface of the final foam product in particular ΔP can be easily measured instrumentally on a continuous basis and an alarm sounded if the value of ΔP ever falls below a given value.

The following steps have been among those found to be effective in correcting for a drift downwards in the value of ΔP . Firstly, the temperature of the mixing device can be reduced by a few degrees centigrade. Secondly, a throttle valve located between the mixer and the die can be partially closed. Thirdly, the blowing agent flow rate can be reduced, thereby increasing the viscosity of the partially mixed polymer and blowing agent. Fourthly, the flow rate of the polymer can be increased (as by increasing the RPM of a gear

ozone depletion in the preparation of foam in accordance with the present invention and eliminate or reduce the concentration of fully halogenated chlorofluorocarbons include: ethyl chloride (EtCl), carbon dioxide (CO₂), chlorodifluoromethane, 1,1-difluoroethane, nitrogen (N₂), water (H₂O), the aliphatic hydrocarbons including, methane, ethane, ethylene, propane, propylene, butane, butylene, isobutane, pentane, neopentane, isopentane, hexane, heptane and mixtures of any of these additional blowing agents.

Particularly useful are methane, ethane, propane, ethyl chloride, carbon dioxide, nitrogen, water and chlorodifluoromethane (CFC-22).

The term "blowing agent" as used in this specification shall refer to both a single blowing agent and mixtures of blowing agents.

The blowing agent usually is present in the process of the present invention at a level of about 3 to about 30 parts by weight per 100 parts by weight of alkenyl aromatic synthetic resin.

Specific blowing agents useful in the process of the present invention for the preparation of alkenyl aromatic synthetic resin foams are (all percents are weight percents based on the total weight of the blowing agent):

(1) CFC-12, CFC-124, CFC-134A, CFC-142B, CFC-143A and mixtures thereof;

(2) Any of the CFCs of 1 in a mixture with up to 6 percent CO₂;

(3) 55 to 97 percent EtCl and 3 to 45 percent CO₂;

(4) The blowing agent of (3) in a mixture with up to 90 percent of a CFC selected from CFC-12, CFC-142B and mixtures thereof;

(5) 19 to 97 percent EtCl and 3 to 81 percent CO₂;

(6) The blowing agent of (5) in a mixture with up to 90 percent of a CFC selected from CFC-12, CFC-142B or mixtures thereof;

(7) The blowing agent of (3) in a mixture with up to 90 percent of a mixture of CFC-12 and one or more CFCs selected from CFC-134A, CFC-124 and CFC-143A;

(8) The blowing agent of (5) in a mixture with up to 90 percent of a mixture of CFC-12 and one or more CFCs selected from CFC-134A, CFC-124 and CFC-143A;

(9) 20 to 97 percent EtCl and 3 to 80 percent of CFC-12, CFC-142B, CFC-134A, CFC-124, CFC-143A and mixtures thereof;

(10) The blowing agent of (9) in a mixture with up to 3 percent CO₂;

(11) The blowing agent of (1) in a mixture with up to 40 percent of CFC-22;

(12) The blowing agent of (11) in a mixture with up to 5.5 percent CO₂;

(13) The blowing agent of (1) in a mixture with up to 50 percent ethane;

(14) The blowing agent of (13) in a mixture with up to 6 percent CO₂;

(15) The blowing agent of (1) in a mixture with up to 50 percent propane;

(16) The blowing agent of (15) in a mixture with up to 6 percent CO₂;

(17) The blowing agent of (5) in a mixture with up to 90 percent of the blowing agent of (1), and up to 50 percent ethane;

(18) The blowing agent of (5) in a mixture with up to 90 percent of the blowing agent of (1), and up to 50 percent propane;

(19) The blowing agent of (5) in a mixture with up to 90 percent of the blowing agent of (1), and up to 50 percent of CFC-22;

(20) CFC-22;

(21) The blowing agent of (20) in a mixture with up to 5 percent CO₂;

(22) The blowing agent of (20) in a mixture with up to 50 percent ethane;

(23) The blowing agent of (21) in a mixture with up to 50 percent ethane;

(24) The blowing agent of (20) in a mixture with up to 50 percent propane;

(25) The blowing agent of (21) in a mixture with up to 50 percent propane;

(26) EtCl and up to 40 percent CO₂;

(27) EtCl and up to 70 percent ethane;

(28) The blowing agent of (26) in a mixture with up to 70 percent ethane;

(29) EtCl and up to 70 percent propane;

(30) The blowing agent of (26) in a mixture with up to 70 percent propane;

(31) EtCl and up to 70 percent CFC-22;

(32) The blowing agent of (26) in a mixture with up to 70 percent CFC-22;

(33) The blowing agent of (31) in a mixture with up to 70 percent ethane;

(34) The blowing agent of (31) in a mixture with up to 70 percent propane;

(35) H₂O;

synthetic resin into an extruder where the resin is heat-plastified.

The heat-plastified resin is then passed through a pressure control device, such as a gear pump. The pressure control device controls the discharge pressure of the extruder and more importantly the inlet pressure to the mixing device, such as a rotary pin mixer.

5 The blowing agent is introduced into the rotary pin mixer and the desired pressure is obtained by adjusting the pressure control device and the temperature of the mixing device.

The discharge from the mixing device is then passed through a cooling device, such as one or more heat exchangers of the variety shown in U.S. Patent No. 3,014,702.

10 The discharge from the cooling device is then passed through the die and expanded. The foam examples in this specification are expanded at atmospheric pressure; however, the foam expansion could also occur in subatmospheric pressure.

By maintaining a constant die inlet pressure and adjusting the pressure drop from the mixer's inlet to the die's inlet over a range of pressure drops such that the quality of the extruded foam's surface changes from poor to good (or vice versa), a "critical minimum pressure drop", ΔP_c , for a given blowing agent can be determined. This critical pressure drop depends on the blowing agent and alkenyl aromatic synthetic resin combination and is easily determined by simple experimentation which consists of holding the die pressure constant while adjusting the mixing device pressure until extruded foam having a good skin and no blow holes is produced with no gassing at the die.

20 The critical pressure drop is then determined at that point and is the difference between the mixing device pressure and the die pressure.

Knowing the critical pressure drop, which is for a given blowing agent and alkenyl aromatic synthetic resin, the die pressure, which must be greater than atmospheric pressure, can be adjusted. However, that die pressure plus the critical pressure drop for that blowing agent must also be greater than the vapor pressure of the blowing agent and is the minimum pressure which must be maintained in the mixing device in order to produce extruded foam having a good skin, virtually no blow holes and little or no gassing at the die.

Restated simply, the sum of the die pressure and the empirically determined critical pressure drop, is the minimum mixing pressure at which the mixing device must be maintained to produce quality extruded alkenyl aromatic synthetic resin foam.

30 The mixing device must be operated at least at the critical mixing pressure and can also be operated above the critical mixing pressure.

This requirement of a minimum operating pressure in the mixing device is not method, process or system dependent; the numerical value of the minimum acceptable operating pressure in the mixing device is primarily dependent on the blowing agent used and much less dependent on the specific extrusion process (such as those shown in Figures 1-19) as well as the precise location of pressure gauges etc. Accordingly, this invention applies to any extrusion method for producing alkenyl aromatic synthetic resin foam.

The following examples illustrate ways in which the principle of the invention has been applied; but should not be construed as limiting the invention.

40 Foams were prepared from several different polymers, a large number of different blowing agents, using apparatus shown schematically in Figure 3. In particular, essentially, a $1\frac{1}{2}$ inch (3.8cm) extruder was used in combination with a $\frac{1}{2}$ horsepower (370W) gear pump manufactured by Zenith; a mixer of the rotary pin type disclosed in U.S. Patent No. 3,770,668; flat plate coolers of the type shown in U.S. Patent 3,014,702; and a slit extrusion die having an adjustable gap. The polymer throughput rate was 10 pounds (4.5kg) per hour. 45 Tables 1-5 show the processing conditions and some of the product properties, as well as the empirically determined values of critical pressure drop, ΔP_c , for each of the exemplified combinations of polymer and blowing agents.

The following abbreviations are employed in this specification, including the drawings:

50	PS	polystyrene having a weight average molecular weight of about 200,000 as measured by the gel permeation chromatograph method
	SAA	styrene/acrylic acid copolymer having a weight average molecular weight of about 165,000 as measured by the gel permeation chromatograph method
	CISAA	calcium ionomer of SAA
	BA	blowing agent
55	pph	parts per hundred
	F	degrees Fahrenheit
	RPM	revolutions per minute

Table 1 LONG-TERM INSULATING BLOWING AGENTS FOR POLYSTYRENE FOAMS

Example No.	Polymer Type	Blowing Agent Systems (Components in (pph))	Total BA Level (pph)	Foam Temp. T_f ($^{\circ}$ F)	Mixer RPM	P_M Inlet Pressure (psi)	T_{Mixer} ($^{\circ}$ C)	P_{Di} Inlet Pressure (psi)	P_{MC} Critical Pressure (psi)	ΔP_C Critical Pressure Drop (psi)	Foam Density (pct)	Foam Cell Size (mm)	Compressive Strength (psi)		Quality of Foam Surface
													MD	TD	
1A 1B	PS	C1C-12	14.4	130	10	1550	150	1090	1530	440	2.46	0.1	---	---	Good
2A 2B	PS	C1C-142B	11.9	130	10	1000	164	1090	1020	370	3.09	1.2	115	59.7	Poor
3A 3B	PS	6.5 C1C-12/ 6.5 C1C-142B	13.0	130	10	1300	173	650	1190	350	3.11	1.8	17.2	7.7	Good
4A 4B 4C 4D	PS	10.0 CFC-12/ 1.1 CO ₂	11.9	130	10	1750	155	840	1660	460	2.42	0.59	22.3	28.0	Good
5A 5B	PS	9.0 C1C-142B/ 1.1 CO ₂	10.1	130	10	1690	159	1210	1520	320	2.28	0.43	36.0	32.1	Poor
6A 6B	PS	C1C-142B/CFC-12/CO ₂ 4.9/4.9/1.1	10.9	130	10	1430	174	1200	1450	480	---	---	---	---	Poor
7A 7B	PS	C1C-22/CO ₂ /CFC-12 4.0/1.3/6.0	11.3	135	10	1370	178	850	1430	500	2.64	0.90	---	---	Poor
8A 8B	PS	C1C-22/CO ₂ /C1C-142B 3.1/1.3/7.0	12.0	130	10	1210	164	910	1100	270	2.46	0.85	12.6	49.3	Good
						1160	159	910			1.82	0.22	---	---	Good
											1.77	0.26	---	---	Poor

TABLE 1 LONG-TERM INSULATING BLOWING AGENTS FOR POLYSTYRENE FOAMS
(CONTINUED)

Example No	Polymer Type	Blowing Agent Systems (Components in ppb)	Total BA Level (ppb)	Foam Temp T_f ($^{\circ}$ F)	Mixer RPM	P_M Inlet Pressure (psi)	T_{Mixer} ($^{\circ}$ C)	P_D Die Inlet Pressure (psi)	P_{MC} Critical Pressure (psi)	ΔP_C Critical Pressure Drop (psi)	Foam Density (pcf)	Foam Cell Size (mm)	Compressive Strength (psi)		Quality of Foam Surface
													MO	TD	
14A 14B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 3.5/1 3/6 0/0 25	11.1	130	10	1120	154	800	1090	290	1.77	0.20	---	---	Good
15A 15B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 3.5/1 3/6 0/0 37	11.2	130	10	1150	179	800	1160	280	1.87	0.59	20.9	32.4	Poor
16A 16B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 3.5/1 3/6 0/0 7	11.5	130	10	1160	170	900	1170	270	1.79	0.46	---	---	Poor
17A 17B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 2.5/1 3/6 0/0 4	10.2	130	10	1000	169	720	1020	300	2.07	0.96	---	---	Poor
18A 18B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 2.5/1 3/6 0/0 6	10.4	130	10	1080	154	800	1050	250	1.73	0.59	26.1	30.3	Good
19A 19B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 2.5/1 3/6 0/0 2	11.0	130	10	890	154	590	880	290	2.11	2.71	10.5	45.9	Good
20A 20B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 1.0/1 3/6 0	8.3	130	10	1140	179	790	1200	410	1.96	2.32	16.8	10.8	Poor
21A 21B	PS	1,1,1- $Cl_3C_3F_3$ /1,2- $Cl_2C_2H_2$ 1.0/1 3/6 5	8.8	130	10	1540	157	1070	1525	455	1.86	0.30	---	---	Good

Table 1 Metric Equivalents
(Continued)

Example No.	Foam Temp TF (°C)	PM Mixer inlet pressure (MPa)	PD Die inlet Pressure (MPa)	PMC Critical Pressure (MPa)	Δ PC Critical Pressure Drop (MPa)	Foam Density (kg/m ³)	Compressive Strength (kPa)	
							MD	TD
9A	54	9.4	6.2	9.4	3.2	35.2	-	-
9B	54	9.5	6.2			34.1	-	-
10A	54	9.8	6.5	9.9	3.4	36.0	-	-
10B	54	10.0	6.5			34.6	-	-
11A	54	10.0	7.4	9.7	2.3	32.5	-	-
11B	54	9.4	7.4			29.8	-	-
12A	54	10.3	7.4	10.1	2.7	34.3	-	-
12B	54	9.8	7.4			33.5	-	-
13A	54	12.8	9.0	12.6	3.7	35.2	-	-
13B	54	12.4	9.0			36.5	-	-

TABLE 2 LONG-TERM INSULATING BLOWING AGENTS FOR SAA (97/3) COPOLYMER FOAMS

Example No	Polymer Type	Blowing Agent Systems (Components in ppb)	Total BA Level (ppb)	Foam Temp T_f ($^{\circ}F$)	Mixer RPM	P_M Mixer Inlet Pressure (psi)	T_{mixer} ($^{\circ}C$)	P_D Die Inlet Pressure (psi)	P_{MC} Critical Pressure (psi)	ΔP_C Critical Pressure Drop (psi)	Foam Density (pcf)	Foam Cell Size (mm)	Compressive Strength (psi)		Quality of Foam Surface
													MD	TD	
22A	SAA (3%AA)	CFC-12/CO ₂	12.6	130	10	1640	182	1110	1690	500	2.50	0.11	---	75.4	Poor
22B	-	11.6/1.0	-	-	-	1730	169	1110	-	-	2.57	0.13	---	77.0	Good
22C	-	-	-	-	-	2040	164	1450*	-	-	2.57	0.22	---	67.0	Excellent
23A	SAA (3%AA)	CFC-142B	11.9	130	10	1170	168	770	1110	340	2.84	0.76	---	63.1	Good
23B	-	-	-	-	-	1110	171	770	-	-	3.18	1.08	---	51.8	Good
23C	-	-	-	-	-	1090	176	770	-	-	2.99	1.08	---	57.3	Poor
23D	-	-	-	-	-	1400	176	1000*	-	-	2.84	1.08	---	55.5	Good
24A	SAA (3%AA)	CFC-142B/CO ₂	10.7	130	10	1210	180	900	1290	390	2.70	0.81	26.0	59.0	Poor
24B	-	9.7/1.0	-	-	-	1350	163	900	-	-	2.70	0.85	28.1	61.7	Good
25A	SAA (3%AA)	CFC-142B/CFC-12	13.0	130	10	1320	164	970	1290	320	2.26	0.19	---	---	Good
25B	-	6.5/6.5	-	-	-	1260	168	970	-	-	2.20	0.30	---	---	Poor
26A	SAA (3%AA)	CFC-142B/CO ₂ /CFC-12	11.6	130	10	1290	161	910	1290*	380	2.74	0.31	---	57.4	Good
26B	-	5.3/1.0/5.3	-	-	-	1250	173	910	-	-	2.25	0.32	---	57.8	Poor

* Pressure was increased by decreasing the die gap.

Table 2 Metric Equivalents

Example No.	Foam Temp T_f ($^{\circ}\text{C}$)	PM Mixer Inlet Pressure (MPa)	PD Die Inlet Pressure (MPa)	PMC Critical Pressure (MPa)	ΔP_C Critical pressure Drop (MPa)	Foam Density (kg/m^3)	Compressive Strength (kPa)	
							MD	TD
22A	54	11.3	7.7	11.7	4.0	41.3	-	520
22B	54	11.9	7.7			41.2	-	531
22C	54	14.1	10.0			41.2	-	462
23A	54	8.1	5.3	7.7	2.3	45.5	-	435
23B	54	7.7	5.3			50.9	-	357
23C	54	7.5	5.3			47.9	-	395
23D	54	9.7	6.9			45.5	-	383
24A	54	8.5	6.2	8.9	2.7	43.2	248	407
24B	54	9.3	6.2			43.2	194	425
25A	54	9.1	6.7	8.9	2.2	36.2	-	-
25B	54	8.7	6.7			36.5	-	-
26A	54	8.9	6.3	8.9	2.6	43.9	-	396
26B	54	8.6	6.3			36.0	-	399

TABLE 3 LONG-TERM INSULATING BLOWING AGENTS FOR CISAA (9/7/3) COPOLYMER FOAMS

Sample No.	Polymer Type	Blowing Agent Systems (Components in ppb)	Total BA Level (ppb)	Foam Temp T_f ($^{\circ}$ F)	Miner RPM	P_{in} Miner Inlet Pressure (psi)	T_{mixer} ($^{\circ}$ C)	PD Die Pressure (psi)	P_{mc} Critical Pressure (psi)	ΔP_c Critical Pressure Drop (psi)	Foam Density (pct)	Foam Cell Size (mm)	Compressive Strength (psi)		Quality of Foam Surface
													MD	ID	
32A	5AA (3% SAA)	CFC-12/CO ₂	12.6	130	10	1610	103	1100	1700	520	2.04	0.10	65.2	65.2	Poor
32B	0.5 ppb Ca(OH) ₂	11.6/1.0	-	-	-	1700	164	1100	-	-	2.67	0.10	70.9	70.9	Good
33A	-	CFC-142B	11.9	130	10	1050	169	750	1010	260	2.12	0.16	43.3	43.3	Good
33B	-	-	-	-	-	990	171	750	-	-	2.12	0.14	39.7	39.7	Poor
34A	-	CFC-142B/CO ₂	10.7	130	10	1320	182	860	1350	490	2.75	0.13	55.2	55.2	Poor
34B	-	9.7/1.0	-	-	-	1380	168	860	-	-	2.06	0.14	60.9	60.9	Good
34C	-	-	-	-	-	1570	166	1040*	-	-	2.62	0.14	75.2	75.2	Excellent
35A	-	CFC-12/CFC-142B	13.0	130	10	1200	178	820	1310	490	2.78	0.11	63.7	63.7	Poor
35B	-	6.5/6.5	-	-	-	1260	167	820	-	-	2.73	0.13	63.7	63.7	Fair
35C	-	-	-	-	-	1720	165	1220	-	-	2.79	0.13	63.7	63.7	Good
36A	-	CFC-142B/CO ₂ /CFC-12	10.9	130	10	2240	161	1680	2200	520	2.06	0.13	63.7	63.7	Good
36B	-	4.9/1.1/4.9	-	-	-	2160	174	1680	-	-	2.83	0.11	63.7	63.7	Poor
37A	-	CFC-22/CO ₂ /CFC-12	13.0	130	10	1950	170	1450	1950	500	2.54	0.11	63.7	63.7	Good
37B	-	4.7/1.3/1.0	-	-	-	1900	175	1450	-	-	2.65	0.11	63.7	63.7	Poor
38A	-	CFC-22/CO ₂ /CFC-142B	12.0	130	10	1570	180	1300	1630	330	2.76	0.13	63.7	63.7	Poor
38B	-	3.7/1.3/1.0	-	-	-	1630	159	1300	-	-	2.32	0.11	63.7	63.7	Good

*Pressure was increased by decreasing the die gap.

Table 3 Metric Equivalents

Example No.	Foam Temp T _F (°C)	PM Mixer inlet Pressure (MPa)	PD Die inlet Pressure (MPa)	PMC Critical Pressure (MPa)	Δ PC Critical Pressure Drop (MPa)	Foam Density (kg/m ³)	Compressive Strength (kPa)	
							MD	TD
32A	54	11.1	8.1	11.7	3.6	42.3	-	450
32B	54	11.7	8.1			42.8	-	489
33A	54	7.2	5.2	7.0	1.8	34.0	-	299
33B	54	6.8	5.2			34.0	-	274
34A	54	9.1	5.9	9.3	3.4	44.1	-	381
34B	54	9.5	5.9			45.8	-	420
34C	54	10.8	7.2			42.0	-	519
35A	54	8.3	5.7	9.0	3.4	44.5	-	439
35B	54	8.7	5.7			43.7	-	-
35C	54	11.9	8.4			44.7	-	-
36A	54	15.4	11.6	15.2	3.6	33.0	-	-
36B	54	14.9	11.6			45.3	-	-
37A	54	13.4	10.0	13.4	3.4	40.7	-	-
37B	54	13.1	10.0			42.4	-	-
38A	54	10.8	9.0	11.2	2.3	36.2	-	-
38B	54	11.2	9.0			37.2	-	-

TABLE 4 NON-LONG-TERM INSULATING BLOWING AGENTS FOR POLYSTYRENE FOAMS

Example No.	Polymer Type	Blowing Agent Systems (Components in ppb)	Total DA level (ppb)	Foam Temp T_f ($^{\circ}$ F)	Mixer RPM	P_{mixer} Inlet Pressure (psi)	T_{mixer} ($^{\circ}$ C)	P_{in} Die Inlet Pressure (psi)	P_{rel} Critical Pressure (psi)	ΔP_c Critical Pressure Drop (psi)	Foam Density (pcf)	Foam Cell Size (mm)	Compressive Strength (psi)		Quality of Foam Surface
													MD	TD	
44A 44B	PS	CFC-22/CO ₂ /C ₃ H ₈ 7.6/1.1/1.4	10.1	130	10	1400 1360	155 162	1030	1380	350	1.90 1.89	0.13 0.13	---	---	Good Poor
45A 45B	PS	CFC-22/CO ₂ /C ₃ H ₈ 6.0/1.3/1.9	9.2	130	10	1250 1220	153 156	890	1230	340	1.86 1.89	0.28 0.30	---	4/3	Good Poor
46A 46B	PS	E14/CO ₂ /C ₃ H ₈ 3.5/1.3/1.7	6.5	130	10	930 950	174 165	660 660	940	280	2.05 2.08	2.71 3.24	10.5 9.7	49.2 52.8	Poor Good
47A 47B	PS	E14/CO ₂ /C ₃ H ₈ 3.5/0.9/2.5	6.9	130	10	1060 1000	155 167	700 700	1030	330	2.41 2.18	2.79 2.0	13.2 15.7	72.8 62.3	Good Poor
48A 48B	PS	E14/CO ₂ /CFC-22 3.5/1.3/5.0	9.8	130	10	1040 1110	180 162	770 770	1070	300	1.95 1.98	3.35 2.80	---	---	Poor Good
49A 49B	PS	E14/CFC-22/C ₃ H ₈ 3.5/5.0/0.9	9.4	130	10	990 1030	177 169	680 680	1010	330	2.29 2.23	1.58 2.86	---	---	Poor Good
50A 50B	PS	E14/CFC-22/C ₃ H ₈ 3.5/5.0/1.3	9.8	130	10	920 890	153 164	590 590	900	310	2.23 2.19	2.72 3.61	7.3 6.3	59.9 58.4	Good Poor

Table 4 Metric Equivalents

Example No.	Foam Temp T _F (°C)	PM Mixer inlet pressure (MPa)	PD Die inlet pressure (MPa)	PMC Critical pressure (MPa)	APC Critical pressure Drop (MPa)	Foam Density (kg/m ³)	Compressive Strength (kPa)	
							MD	TD
44A	54	9.7	7.1	9.5	2.4	30.4	-	-
44B	54	9.4	7.1			30.3	-	-
45A	54	8.6	6.1	8.5	2.3	29.8	-	-
45B	54	8.4	6.1			30.3	167	326
46A	54	6.4	4.6	6.5	1.9	32.8	72	339
46B	54	6.6	4.6			33.3	67	364
47A	54	7.3	4.8	7.1	2.3	38.6	91	502
47B	54	6.9	4.8			34.9	108	430
48A	54	7.2	5.3	7.4	2.1	31.2	-	-
48B	54	7.7	5.3			31.7	-	-
49A	54	6.8	4.7	7.0	2.3	36.7	-	-
49B	54	7.1	4.7			35.7	-	-
50A	54	6.3	4.1	6.2	2.1	35.7	50	413
50B	54	6.1	4.1			35.1	43	403

foregoing is intended to be merely illustrative and is not to be construed or interpreted as being restrictive or otherwise limiting of the present invention, excepting as it is set forth and defined in the hereto-appended claims.

TABLE 5 NON-LONG-TERM INSULATING BLOWING AGENTS FOR SAA AND CISAA COPOLYMER FOAMS

Example No	Polymer type	Blowing Agent Systems (Components in ph)	Total BA Level (pph)	Foam Temp T_f ($^{\circ}$ F)	Mixer (RPM)	P_M Inlet Pressure (psi)	T_{mixer} ($^{\circ}$ C)	P_D Die Inlet Pressure (psi)	P_{ac} Critical Pressure (psi)	ΔP_c Critical Pressure Drop (psi)	Foam Density (pct)	Foam Cell Size (mm)	Compressive Strength (psi)		Quality of foam Surface
													MD	TD	
54A 54B	SAA (3%AA)	EtCl/CO ₂ /C ₂ H ₆ 3.5/1.3/1.7	6.5	130	10	1360 1310	164 170	940 940	1340	400	2.72 2.63	0.54 0.32	---	40.1 ---	Good Poor
55A 55B	SAA (3%AA)	H ₂ O/CO ₂ /C ₂ H ₆ 1.0/1.3/2.5	4.8	130	10	1560 1480	165 168	1150 1150	1520	370	2.47 2.43	0.38 0.28	---	---	Good Poor
56A 56B	SAA (3%AA)	H ₂ O/CO ₂ /EtCl 0.97/1.3/3.7	6.0	130	10	1560 1590	180 171	1060 1060	1570	510	2.53 2.70	3.25 2.17	12.5 20.0	30.6 35.7	Poor Good
57A 57B	SAA (3%AA)	H ₂ O/CO ₂ /CFC-22 1.0/1.63/6.12	8.75	130	10	1710 1880	180 166	1360 1360	1790	430	2.21 2.18	0.93 0.46	---	39.1 31.7	Poor Good
58A 58B	SAA (3%AA) 0.5 pph Ca(OH) ₂	EtCl/CO ₂ /C ₂ H ₆ 3.5/1.3/1.7	6.5	130	10	1570 1400	166 179	850 850	1480	630	3.16 2.91	0.24 0.19	73.1 ---	49.5 36.9	Good Poor

Table 5 Metric Equivalents

Example No.	Foam Temp T_f ($^{\circ}\text{C}$)	PM Mixer inlet Pressure (MPa)	PD Die inlet Pressure (MPa)	PMC Critical Pressure (MPa)	Δ PC Critical Pressure Drop (MPa)	Foam Density (kg/m^3)	Compressive Strength (kPa)	
							HD	TD
54A	54	9.4	6.5	9.2	2.8	43.6	-	276
54B	54	9.0	6.5			42.1	-	-
55A	54	10.8	7.9	10.5	2.6	39.6	-	-
55B	54	10.2	7.9			38.9	-	-
56A	54	10.8	7.3	10.8	3.5	40.5	86	211
56B	54	11.0	7.3			43.2	138	246
57A	54	11.8	9.4	12.3	3.0	35.4	-	270
57B	54	13.0	9.4			34.9	-	219
58A	54	10.8	5.9	10.2	4.3	50.6	504	341
58B	54	9.7	5.9			46.6	-	254
59A	54	9.6	7.0	9.4	2.4	40.8	-	-
59B	54	9.3	7.0			42.6	-	-
60A	54	11.0	7.6			44.1	340	474
60B	54	7.7	7.7			40.2	228	288
60C	54	8.4	5.0	8.4	3.4	42.3	345	308

55 Claims

1. A method for producing a thermoplastic

extruded foam body having closed cells including the steps of:

- (22) A blowing agent of (20) including up to 50 percent ethane;
 (23) A blowing agent of (21) including up to 50 percent ethane;
 (24) A blowing agent of (20) including up to 50 percent propane;
 (25) A blowing agent of (21) including up to 50 percent propane;
 5 (26) 20 to 90 percent EtCl and up to 40 percent CO₂;
 (27) 20 to 90 percent EtCl and up to 70 percent ethane;
 (28) A blowing agent of (26) including up to 70 percent ethane;
 (29) 20 to 90 percent EtCl and up to 70 percent propane;
 (30) A blowing agent of (26) including up to 70 percent propane;
 10 (31) 20 to 90 percent EtCl and up to 70 percent CFC-22;
 (32) A blowing agent of (26) including up to 70 percent CFC-22;
 (33) A blowing agent of (31) including up to 70 percent ethane;
 (34) A blowing agent of (31) including up to 70 percent propane;
 (35) H₂O;
 15 (36) 0.4 percent to 99.9 percent H₂O and 0.1 percent to 50 percent CO₂;
 (37) A blowing agent of (36) including up to 99.5 percent of the blowing agent of (1);
 (38) 0.4 to 99.9 percent H₂O and up to 60 percent CFC-22;
 (39) A blowing agent of (36) including up to 60 percent CFC-22;
 (40) A blowing agent of (38) including up to 60 percent of ethane, propane, EtCl or mixtures thereof;
 20 (41) 0.4 to 99.9 percent H₂O and up to 60 percent ethane;
 (42) A blowing agent of (36) including up to 60 percent ethane;
 (43) 0.4 to 99.9 percent H₂O and up to 60 percent propane;
 (44) A blowing agent of (36) including up to 60 percent propane;
 (45) 0.4 to 99.9 percent H₂O and up to 60 percent EtCl; and
 25 (46) A blowing agent of (36) including up to 60 percent EtCl.
4. A method as claimed in any one of the preceding claims wherein the resin is polystyrene.
5. A method as claimed in any one of Claims 1, 2 and 3, wherein the resin is a styrene/ acrylic acid
 30 copolymer having one tenth (0.1) weight percent to fifteen (15) weight percent polymerized acrylic acid
 by total resin weight.
6. A method as claimed in any one of Claims 1, 2, 3 and 5, wherein the resin is an ionomeric
 styrene/acrylic acid copolymer.
- 35 7. A method as claimed in Claim 6, wherein the ion is selected from calcium, sodium, lithium, potassium,
 magnesium and mixtures of these ions.
8. A method as claimed in Claim 7, wherein the ions for the ionomeric styrene/acrylic acid copolymer are
 40 provided by addition to the heat plastified resin in step (a) of one tenth (0.1) to one (1) parts per
 hundred by weight per hundred parts by weight of resin of a neutralizing agent selected from calcium
 hydroxide, lithium hydroxide, sodium hydroxide, potassium hydroxide, magnesium oxide and mixtures
 of these compounds.
- 45 9. A method as claimed in Claim 8, wherein said amount of neutralizing agent added is one tenth (0.1) to
 six tenths (0.6) parts per hundred by weight per hundred parts by weight of resin.
10. A method as claimed in any one of the preceding claims further comprising the step of passing the
 plastified resin through a pressure control device, after step (a) and before step (b).
- 50 11. A method as claimed in any one of the preceding claims, wherein a drop in the value of ΔP is
 corrected by
 (a) reducing the temperature of the mixing device;
 (b) partially closing a throttle valve located between the mixing device and the die's inlet;
 55 (c) reducing the blowing agent concentration; and/or
 (d) increasing the feed rate of the resin into the mixing device.
12. A method as claimed in any one of the preceding claims wherein the value of ΔP is continuously

